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DOC. NO.
NARF-62-3T
MR-N-292

EFFECTS OF REACTOR RADIATION ON THE PROPERTIES OF MIX BIS (PHENOXYPHENYL) ETHER OPERATING IN A HYDRAULIC PUMP LOOP

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NUCLEAR AEROSPACE RESEARCH FACILITY

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GENERAL DYNAMICS | FORT WORTH

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30 MARCH 1962

EFFECTS OF REACTOR RADIATION ON THE PROPERTIES OF MIX BIS (PHENOXYPHENYL) ETHER OPERATING IN A HYDRAULIC PUMP LOOP

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SECTION II, TASK III, ITEM 4
OF FZM-2386

CONTRACT
AF 33(657)-7201

ISSUED BY THE
ENGINEERING
DEPARTMENT

GENERAL DYNAMICS | FORT WORTH

ABSTRACT

An experimental hydraulic fluid, mix bis (phenoxyphenyl) ether, was irradiated under operational environmental conditions imposed by a hydraulic pump loop. The Ground Test Reactor at General Dynamics/Fort Worth was used to provide a mixed neutron-gamma radiation field.

The fluid was exposed to an average dose of 2.7×10^{10} ergs/gm(C) of gamma radiation and 3.9×10^{15} nvt of neutrons ($E_n > 2.9$ Mev) Pump-loop conditions during irradiation were 3000 psi and 4-gpm flow. Bulk-oil temperature ranged from 326 to 340°F.

A preirradiation control evaluation was conducted with the pump loop operating 9.75 hours and bulk-oil temperature ranging from 350 to 380°F.

The results of property and performance tests conducted on samples of the fluid during irradiation were compared to data on the nonirradiated fluid to assess the radiation damage. All fluid testing was accomplished in the GD/FW Irradiated Materials Laboratory.

REPORT SUMMARY

This report describes the irradiation and testing of an experimental hydraulic fluid, mix bis (phenoxyphenyl) ether. The irradiation was conducted with the Ground Test Reactor at General Dynamics/Fort Worth, in September 1961; fluid testing and analysis were completed in the following November. The abbreviated nomenclature for this fluid is Mixed-4P3E. The fluid was irradiated while being used as the working fluid in a hydraulic pump loop under 3000-psi pump outlet pressure. Fluid flow rate was maintained at approximately 4 gpm and temperature between 326 and 340°F.

The pump loop operated 15.9 hours, with the reactor at power for 11.2 hours of this time. A total gamma dose at 2.7×10^{10} ergs/gm(C) and a neutron exposure of 3.9×10^{15} n/cm^2 ($E_n > 2.9$ Mev) were accumulated by the fluid. Samples of the fluid were withdrawn for testing after 5.0 and 10 hours of irradiation.

A preirradiation control evaluation was conducted in the Irradiated Materials Laboratory with pump-loop conditions of 3000 psi, 4 gpm, and a bulk-oil temperature of between 350 and 380°F. Test samples were withdrawn after 2.5, 5.0, and 9.75 hours (final) of loop operation.

A comparison of the properties and performance characteristics of the irradiated samples with similar data on the "as received" fluid and the fluid from the control evaluation indicated the extent of radiation damage.

The Mixed-4P3E suffered only minor changes in properties and performance characteristics during irradiation in the hydraulic pump loop; therefore, the conditions imposed in this experiment did not prove to be limiting conditions for this type of fluid. The data do indicate, however, several manifestations of radiation damage, any of which might become a limiting criterion at some higher radiation dose, temperature, or more severe combination of these environments Important in this respect were an increase in viscosity, broadening of the boiling range, the formation of insolubles, decreased initial thermal decomposition temperature, a loss in oxidation resistance, and a loss in boundary lubrication ability.

Future work at GD/FW will include the irradiation of higher molecular weight homologs of the 4P3E fluid type. Irradiations are anticipated for these fluids in both hydraulic and lubricating applications.

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I. INTRODUCTION

Damage to materials resulting from exposure to nuclear radiation is being investigated at General Dynamics/Fort Worth (GD/FW). This work includes an effort to determine which materials are most suitable for use in environments combining high temperature, mechanical shear stress, and nuclear radiation.

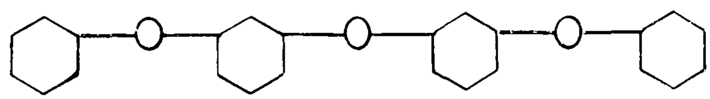
One phase of this work has been directed toward the evaluation of conventional and experimental hydraulic fluids. Accordingly, an experimental fluid, mix bis (phenoxyphenyl) ether, was irradiated in September 1961, with testing and analysis being completed in the following November. The abbreviated nomenclature for this fluid, Mixed-4P3E, is used throughout this report. Some of the data accumulated in this experiment were published in summary form in Reference 1.

The Ground Test Reactor (GTR) served as the mixed-field radiation source. A simple hydraulic test loop was used to maintain temperature and mechanical shear conditions in the Mixed-4P3E. A laboratory run, made prior to irradiation, established the mechanical integrity of the test loop. Remote-sampling techniques allowed fluid samples to be withdrawn periodically during the irradiation and during the laboratory run. These samples and a sample of the Mixed-4P3E in the "as received" condition were subjected to a series of property and performance tests which experience has shown will detect damage in hydraulic fluids. All fluid tests were conducted in the GD/FW Irradiated Materials Laboratory (IML).

II. DESCRIPTION OF FLUID AND MAJOR EQUIPMENT

2.1 Mixed-4P3E, or Mix Bis (Phenoxyphenyl) Ether

The abbreviated name, Mixed-4P3E, implies a material of mixed <u>meta</u> and <u>para</u> (with possibly some <u>ortho</u>) isomers of bis (phenoxyphenyl) ether. As indicated by the name, the material structure contains four phenyl groups connected by three ether linkages. The meta isomer has the following structure:



The Mixed-4P3E used in this experiment was supplied by ASD under the code name GTO 927. Mixed-4P3E is representative of a class of materials generally known as the polyphenyl ethers. These materials are recognized to have superior resistance to oxidation, thermal stress, and exposure to nuclear radiation when compared to more conventional petroleum and synthetic-ester-based hydraulic fluids. The polyphenyl ethers have limited low-temperature capabilities, however, with relatively high pour points. Mixed-4P3E, which has a pour point of 20°F, is estimated to have a viscosity-limited pumpability temperature of about 38°F in a hydraulic application. The high-temperature properties are excellent, with initial thermal decomposition at approximately 837°F.

2.2 Hydraulic Pump Loop

A simple hydraulic pump loop was constructed at GD/FW for use in this experiment. The device provided a means of imposing thermal and mechanical shear stress on the fluid during irradiation. Figure 1 is a block diagram of the pump loop. The assembled loop is shown in Figure 2. An aluminum reservoir contained about 85% (2.5 gal) of the test fluid. The other major loop components are listed below.

Hydraulic Pump: Vickers Model PF 36-3909, 20ZE2, fixed 0.251 in.^{3}/rev., 3750 rpm, 3000 psi.

Hydraulic Pressure Relief Valve: Partex Mfg. Corp., Model No. HPLV-A1, AN No. 6279-8CD, range of from 2300 to 4500 psi.

Filter-Hydraulic Line Type: Aircraft Porous Media, Inc., Model No. AC-712-12-1, 10 microns 3000 psi.

Valve common, hand-load setting.

Heat Exchanger: oil to water.

Reservoir, Static: a 1.5-gallon aluminum container. The fluid in this reservoir was completely separated from that in the circulating loop, and was equipped with thermocouples and fluid sampling lines.

The pump loop is a 3000-psi hydraulic assembly with a fluid flow rate of about four gallons per minute. Pressure and temperature were monitored at points shown in Figure 1. The loop was designed to maintain a bulk-oil temperature of 350°F. Mechanical-energy dissipation as heat was sufficient to bring the fluid to temperature, where it was controlled by use of a liquid-to-liquid heat exchanger.

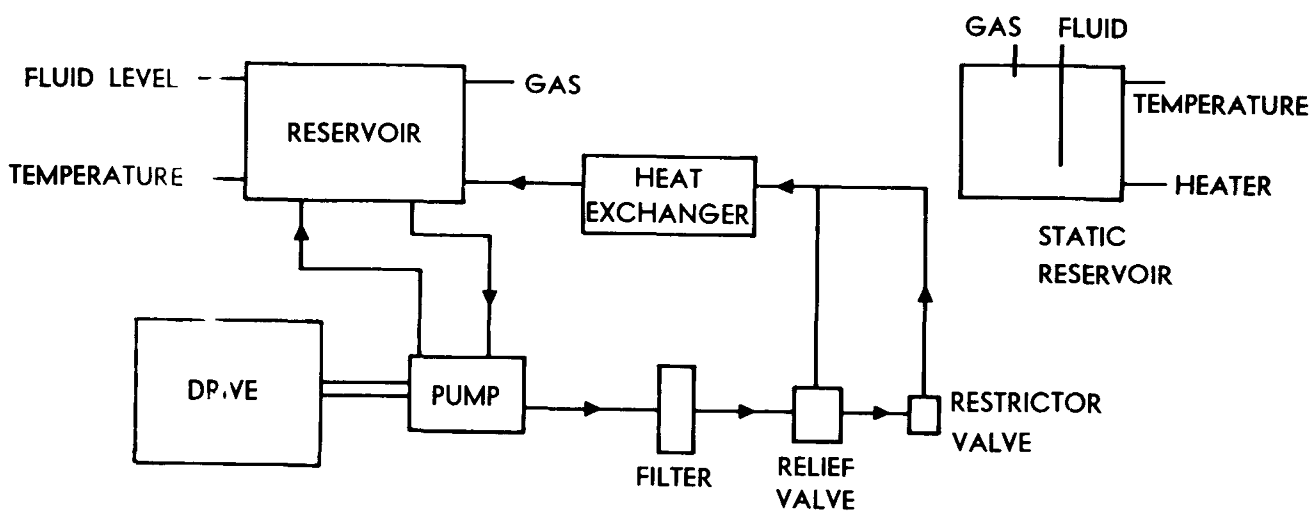


FIGURE 1. BLOCK DIAGRAM OF HYDRAULIC PUMP LOOP

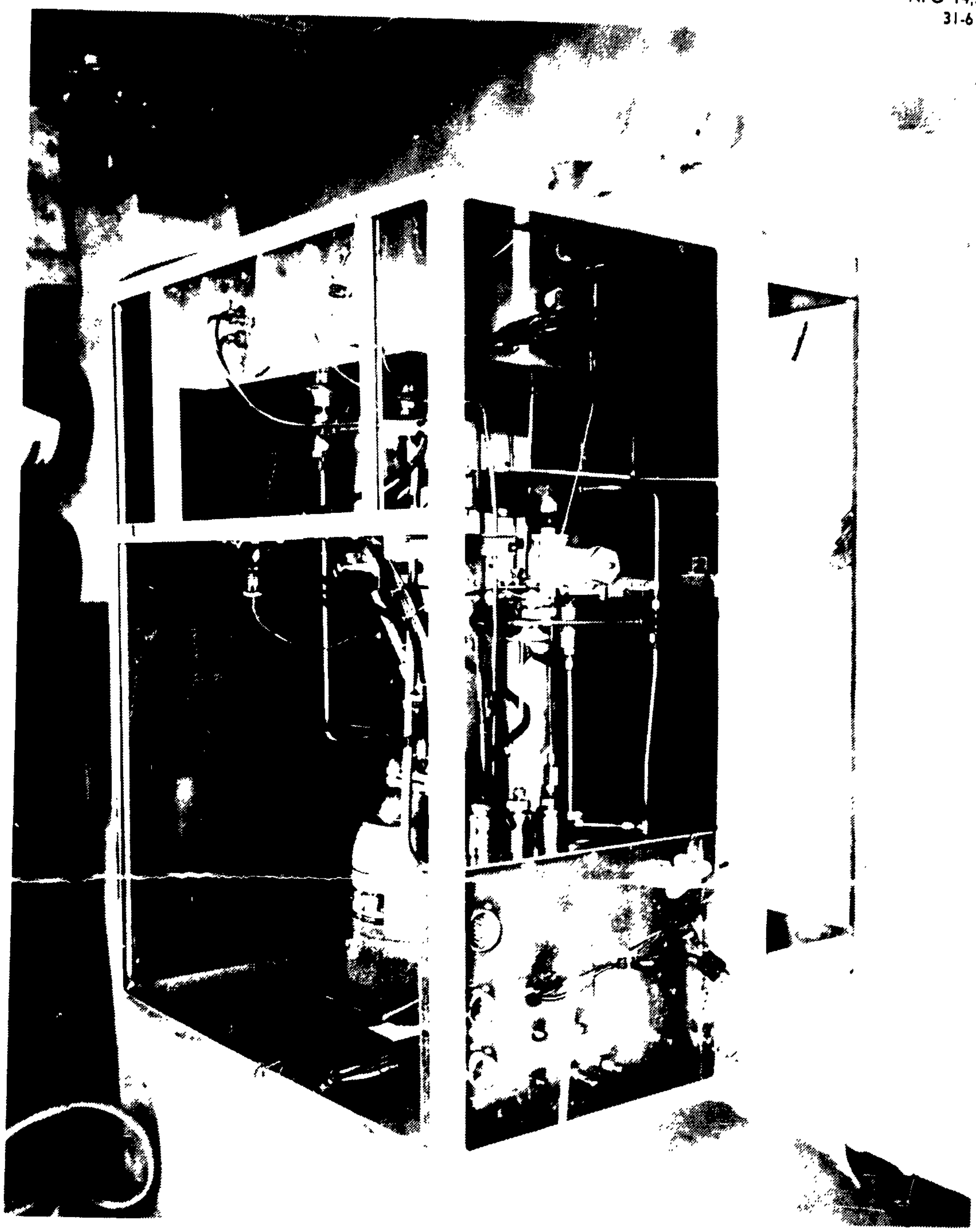


FIGURE 2. HYDRAULIC PUMP LOOP

2.3 Reactor Source and Facilities

2.3.1 Ground Test Reactor

The GTR is a 3-megawatt water-cooled, water-moderated, thermal reactor utilizing MTR-type fuel elements. It is used as the primary source for radiation-effects testing at GD/FW.

2.3.2 Materials Transport System

The irradiation of materials and assemblies using the GTR is accommodated by means of a shuttle system, which transports the test items into and out of the high-flux region. This system permits the irradiation of assemblies weighing up to one ton. Assembly dimensions are limited to an exposure face of approximately 3 x 3 feet and a length of 5 feet. The GD/FW radiation-effects test facilities are described in detail in Reference 2. Figure 3 presents a plan view of the overall transport and reactor system, showing also a test assembly slightly smaller than the hydraulic test loop used for the irradiation of Mixed-4P3E.

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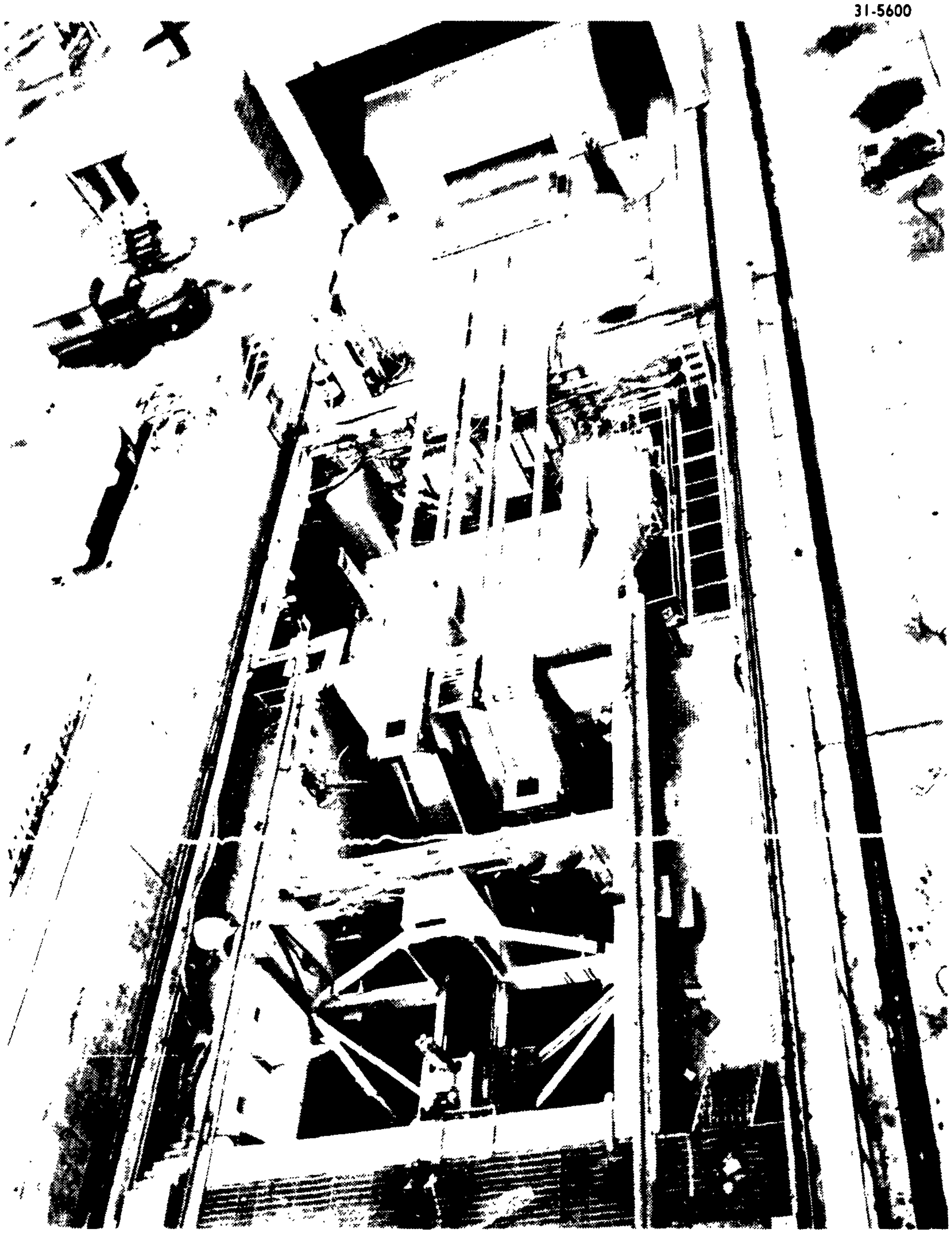

FIGURE 3. MATERIALS TRANSPORT AND REACTOR SYSTEM

III EXPERIMENTAL PROCEDURES

3.1 Control Evaluation

Prior to the irradiation, a control evaluation of Mixed-4P3E fluid was performed in the Irradiated Materials Laboratory for the purpose of determining the effects of dynamic operation on the fluid under normal conditions Hydraulic pump-loop conditions for this evaluation consisted of 3000-psi of fluid pressure, a pumping rate of 4 gpm and a fluid temperature of from 350 to 380°F The control evaluation scheduled for 25 hours was terminated after 9.75 hours because of pump failure. Examination of the pump showed that the universal linkage was worn and that the pin bearing had failed These failures resulted in damage to both the cylinder block and the elastomeric shaft seal. Pump failure was not unexpected, however since the pump is not designed to operate for long periods of time above 300°F; but a calculated risk was taken because the fluid data were needed. Samples of the Mixed-4P3E were taken at the end of 2.5, 5.0, and 9 75 hours of loop operation. Test data on these samples are tabulated and discussed in Section V.

3.2 The Irradiation Evaluation

As a result of the pump failure during the control evaluation, it was decided to maintain the fluid temperature during the irradiation evaluation below 350°F. The pump loop was reconditioned, flushed, and refilled with new Mixed-4P3E for the irradiation. The loop was mounted on the north pallet of the transport system, instrumented to the GTR control room, and exposed to 28.74 megawatt-hours of irradiation. Fluid temperature ranged from 326 to 340°F. This irradiation resulted in a total hydraulic pump-loop operating time of 15.9 hours. This evaluation, though originally scheduled for 25 hours, was terminated prematurely because of pump failure. However, the radiation exposure of the fluid was sufficient to provide a good evaluation of the effects of radiation. Of the 15.9 hours of loop operation time, the reactor was at power for 11.2 hours. Samples of Mixed-4P3E were taken at the end of five hours and ten hours of irradiation. Radiation dose information and test data on these samples are tabulated and discussed in Section V.

3.3 Radiation Dose and Flux Measurements

Packets of radiation detectors were mounted at several points on the hydraulic pump loop for the purpose of measuring the total gamma-dose and neutron-flux exposure of the Mixed-4P3E. Five detector packets were concentrated about the reservoir assembly with three more located on the interior components. Each packet contained one each of the following detectors:

- Nitrous Oxide - Integrated Gamma Dosimeter
- Sulfur Tablet - Fast-Neutron Detector (effective threshold 2.9 Mev)

The details of nitrous-oxide gamma dosimetry have been described in Reference 3. Standard foil-counting procedures were used for the neutron measurements. Neutron data reduction was accomplished during the regularly scheduled IBM 704 computer program. The neutron flux in energy regions above or below that reported can be estimated by consulting the spectrum information contained in Reference 4.

To obtain the final dose and flux values listed in this report (Sec. V, Tables II-VII), the data from the five detectors located about the fluid reservoir were averaged with one calculated point (center point on reservoir face) and one additional point representing the interior components. The center point dose was calculated in lieu of a measurement, because it was not physically possible to place a dosimeter packet between the reservoir and the reactor closet. This procedure provided data points in a number that was approximately proportional to the distribution of the fluid throughout the system; i.e., about six parts of fluid were contained in the reservoir for each part in the other components. Continuous circulation is considered to have distributed the dose equally to all portions of the test fluid.

With the data treatment described above, the Mixed-4P3E that had been irradiated for 10 hours was exposed to a total gamma dose of 2.7×10^{10} ergs/gm(C). The associated neutron exposure was 3.9×10^{15} n/cm^2 considering neutron energies above 2.9 Mev. The sample taken after five hours of irradiation received 1.3×10^{10} ergs/gm(C) and 1.9×10^{15} n/cm^2.

IV. FLUID TESTS AND METHODS

Table I lists the property and performance tests conducted on the various samples of Mixed-4P3E. The method used is listed opposite each test. Wherever possible, testing was in accordance with published procedures, and standard equipment was used.

Some testing was necessary for which standard methods are either not yet available or not yet published in detail. An isoteniscope was constructed which made vapor pressure measurements up to about 900°F possible. Special vapor baths and a wide-range viscometer were used to determine viscosity at 490°F and 704°F. Furthermore, the measurement of neutralization number included a special titration to detect the buildup of phenols. A description of the equipment and procedures used in these special tests is included in the Appendix.

TABLE I. FLUID TESTS AND METHODS FOR MIXED-4P3E

TEST	METHOD
Kinematic Viscosity - - - - - - - -	ASTM D 445-53T (Measurements at 77, 100, 210, 400, 490,* and 704°F*)
Neutralization Number - - - - - - -	ASTM D-664-54 and Special Titration for Phenols*
Flash and Fire Points (COC) - - - -	ASTM D-92-57
Pour Point - - - - - - - - - - - -	ASTM D-91-57
Autogenous Ignition - - - - - - - - Temperature	ASTM D-286-58T
Density @ 60°F - - - - - - - - - -	ASTM D-1217-54
Filtration - - - - - - - - - - - -	Gravimetric Determination Using Millipore Filters
Oxidation and Corrosion - - - - - - Stability	MIL-H-8446B, Sec. 4.5.1
Lubricating Properties - - - - - -	Shell 4-Ball Wear (1-hr runs, 600 rpm, 10 and 50 kg Loads)
Vacuum Distillation - - - - - - - -	ASTM D-1160 (Modified for 100-ml sample size)
Vapor Pressure - - - - - - - - - -	Special Isoteniscope Method (Measurements of from approximately 550 to 900°F)*

* Special equipment and methods used for these tests are described in Appendix A.

V. TEST RESULTS

The Mixed-4P3E irradiated and tested in this experiment suffered only minor changes in properties and performance characteristics. The complete data from all testing are listed in Tables II through VII. The results are summarized below.

5.1 Viscosity-Temperature Characteristics

V-T curves for Mixed-4P3E are shown in Figure 4. Viscosity measurements made over the range of from 77 to 704°F indicate that the fluid was thickened as a result of irradiation. This change was undetectable at temperatures of 400°F and above. The pour point did not change measurably and the viscosity temperature characteristics are not considered to have been damaged appreciably.

5.2 Neutralization Number

Both the ASTM neutralization number and the neutralization number calculated from the special titration for very weak acids (phenols) increased steadily during the irradiation. The maximum neutralization numbers reached, however, were sufficiently low to be considered noncritical.

5.3 Flash Point

Although some flashes were encountered at relatively low temperatures, no values acceptable under the ASTM test method were obtained more than 5°F below the control values.

TABLE II. EFFECTS OF DYNAMIC OPERATION AND REACTOR IRRADIATION ON THE FLUID PROPERTIES OF MIXED-4P3E

CONDITIONS AND TESTS	CONTROL (As-Received)	LABORATORY RUN (Bulk Oil 350-380°F)			IRRADIATION RUN (Bulk Oil 326-340°F)	
Loop Operation Time (hours)	0	2.5	5	9.75	*5	*10
Gamma Exposure [ergs/gm(C)]	0	0	0	0	1.3×10^{10}	2.7×10^{10}
Neutron Exposure [n/cm^2 ($E_n > 2.9$ Mev)]	0	0	0	0	1.9×10^{15}	3.9×10^{15}
Kinematic Viscosity (cs):						
@ 77°F	199.85			186.12		230.31
@ 100°F	69.79	68.22	69.17	68.39	70.00	78.95
@ 210°F	6.32	6.21	6.19	6.21	6.28	6.62
@ 400°F	1.40			1.35		1.40
@ 490°F **	0.93			0.91		0.95
@ 704°F **	0.47			0.46		0.47
Neutralization Number (mgm KOH/gm):						
ASTM D-664-58	0.007	0.011	0.011	0.028	0.082	0.152
Special Titration for Phenols**	Nil	Nil	Nil	Nil	0.086	0.102
Pour Point (°F)	20			20		20
Flash Point (°F)	490	480	490	500	485	485
Fire Point (°F)	555	555	545	545	555	550
Autogenous Ignition Point (°F)	1135			1135		1135
Density (gm/cc @ 60°F)	1.179			1.177		1.177
Filtration [particles > 5.0 μ (mgm/100 ml)]	0.60			28.10***		14.64

* Short periods of time when reactor was not at power are not included.
** See Appendix A for details of this test.
*** Metal particles visible on filter pad

TABLE III. EFFECTS OF DYNAMIC OPERATION AND REACTOR IRRADIATION ON THE OXIDATION AND CORROSION STABILITY OF MIXED-4P3E

Conditions and Tests	Control	Laboratory Run	Irradiation Run
Loop Operation Time (hours)	0	9.75	10
Gamma Exposure [ergs/gm(C)]	0	0	2.7×10^{10}
Neutron Exposure [n/cm^2 (E_n > 2.9 Mev)]	0	0	3.9×10^{15}
Oxidation and Corrosion Stability; 72 hours @ 400°F per MIL-H-8446B:			
Neutralization number after O/C:			
ASTM D-664-58	0.023	0.085	0.492
Special Titration for Phenols	0.005	0.034	0.528
Neutralization number change during O/C:			
ASTM D-664-58	+0.016	+0.057	+0.340
Special Titration for Phenols	+0.005	+0.034	+0.426
Viscosity after O/C (cs @ 210°F)	6.28	6.35	7.85
Viscosity Change During O/C (% @ 210°F)	-0.63	+2.25	+18.58
Weight Change - Metal Specimens (mgm/cm^2):			
Copper	-0.01	-0.10	-0.38
Steel	+0.02	+0.04	+0.01
Silver-Plated Steel	+0.03	+0.03	+0.02
Aluminum	+0.04	+0.03	+0.03
Appearance of Metal Specimens			
Copper	OK	OK	Stained & pitted
Steel	OK	OK	OK
Silver-Plated Steel	OK	OK	OK
Aluminum	OK	OK	OK
Fluid Evaporation During O/C	0	0	0
Sediment Formed	None	None	None
Fluid Appearance After O/C (visual)	Straw	Lt Brown	Dk Brown

TABLE IV. EFFECTS OF DYNAMIC OPERATION AND REACTOR IRRADIATION ON THE LUBRICATING PROPERTIES OF MIXED-4P3E

Conditions and Tests	Control	Laboratory Run	Irradiation Run
Loop Operation Time (hours)	0	9.75	10
Gamma Exposure [ergs/gm(C)]	0	0	2.7×10^{10}
Neutron Exposure [n/cm^2 (E_n > 2.9 Mev)]	0	0	3.9×10^{15}
Shell 4-Ball Wear (1-hr runs @ 600 rpm, 400°F, 52100 1/2-in.-diam Steel balls)			
10 kg load:			
Avg scar diam (mm)	0.775	0.771	0.805
Coefficient of friction (μ)*	0.123	0.121	0.134
50 kg load:			
Avg scar diam (mm)	1.12	1.07	1.19
Coefficient of friction (μ)**	>0.054	>0.054	>0.054

* Represents an average μ for measurements made at 10-minute intervals during each run.

** 0.054 is the maximum μ measurable under a 50-Kg load with the test equipment employed.

TABLE V. ASTM VACUUM DISTILLATION DATA ON IRRADIATED AND NONIRRADIATED MIXED-4P3E

Conditions and Tests	Control (As-Received)	Irradiated (10 hours)
Gamma Exposure [ergs/gm(C)]	0	2.7×10^{10}
Neutron Exposure [n/cm^2(E_n > 2.9 Mev)]	0	3.9×10^{15}
ASTM Vacuum Distillation (ASTM D-1160 Modified for 100-ml sample)	Temp (°F)	Temp (°F)
Distillation @ 10 mm Hg:		
% Recovery = 5	500	452
10	524	510
20	531	531
30	532	531
40	532	531
50	532	531
60	532	532
70	532	532
80	533	534
90	534	(85%) 534
95	534	(88%) 536
97	536	
End Point due to Cracking	(97%) 540	(88%) 540

TABLE VI. VAPOR PRESSURE-TEMPERATURE DATA ON NONIRRADIATED MIXED-4P3E (SPECIAL ISOTENISCOPE)

Control (As-Received)				Control (9.75-hr Loop Operation)			
RUN-1		RUN-2		RUN 1		RUN 2	
Temp (°F)	V.P. (mm Hg)	Temp (°F)	V.P. (mm Hg)	Temp (°F)	V.P. (mm Hg)	Temp (°F)	V.P. (mm Hg)
542	7.5	530	5.5	548	8.5	535	6.7
613	15.0	558	11.0	620	32.2	554	10.2
650	50.0	605	25.5	678	76.7	568	15.0
677	75.0	650	51.0	708	219.0	615	30.7
708	125.5	680	80.0	733	175.5	680	75.2
725	150.5	710	125.2	765	250.0	718	130.9
748	199.5	728	155.5	793	325.0	740	180.0
765	256.5	750	201.0	806	382.0	769	251.4
780	304.5	767	249.5	820	450.3	799	350.4
794	351.5	782	301.0	830	499.6	823	456.6
799	382.0	795	351.5	842	547.5	833	501.2
805	402.5	805	402.5	852	606.0	838	526.4
809	426.0	818	450.0	860	654.0	845	551.0
818	478.0	829	501.0	871	737.5	850	599.0
826	501.0	838	551.5	878	797.8	860	653.0
831	526.0	843	602.0			873	751.5
836	560.0	852	655.5			803	800.0
841	587.0	860	704.0				
851	640.0	867	751.0				
860	705.5	880	800.0				
867	754.5						

TABLE VII. VAPOR PRESSURE-TEMPERATURE DATA ON IRRADIATED MIXED-4P3E (SPECIAL ISOTENISCOPE)

Gamma Exposure: 2.7×10^{10} ergs/gm(C)
Neutron Exposure: 3.9×10^{15} n/cm^2 ($E_n > 2.9$ Mev)
Loop Operation During Irradiation: 10 hours

RUN 1*		RUN 2		RUN 3	
Temp (°F)	V.P. (mm Hg)	Temp (°F)	V.P. (mm Hg)	Temp (°F)	V.P. (mm Hg)
550	14.0	536	8.2	535	8.4
603	30.0	589	23.2	600	30.1
650	60.5	638	52.0	658	75.3
695	101.0	663	76.4	702	118.0
729	153.5	695	110.0	730	170.0
745	179.5	725	165.1	762	249.8
767	225.0	742	200.8	785	325.0
779	250.0	760	250.2	800	379.0
798	300.0	782	327.1	814	450.5
817	351.0	798	379.9	820	498.4
832	401.0	815	449.7	835	550.0
845	523.0	823	500.9	843	600.5
849	645.0	837	596.7	852	652.0
867	700.0	847	654.1	858	697.0
890	800.0	858	705.4	868	749.0
		868	769.5	878	800.0
		873	799.1		

* An additional system pump-down was required during this run after measurements had begun. This was due to an inadvertent pressure increase from the nitrogen supply.

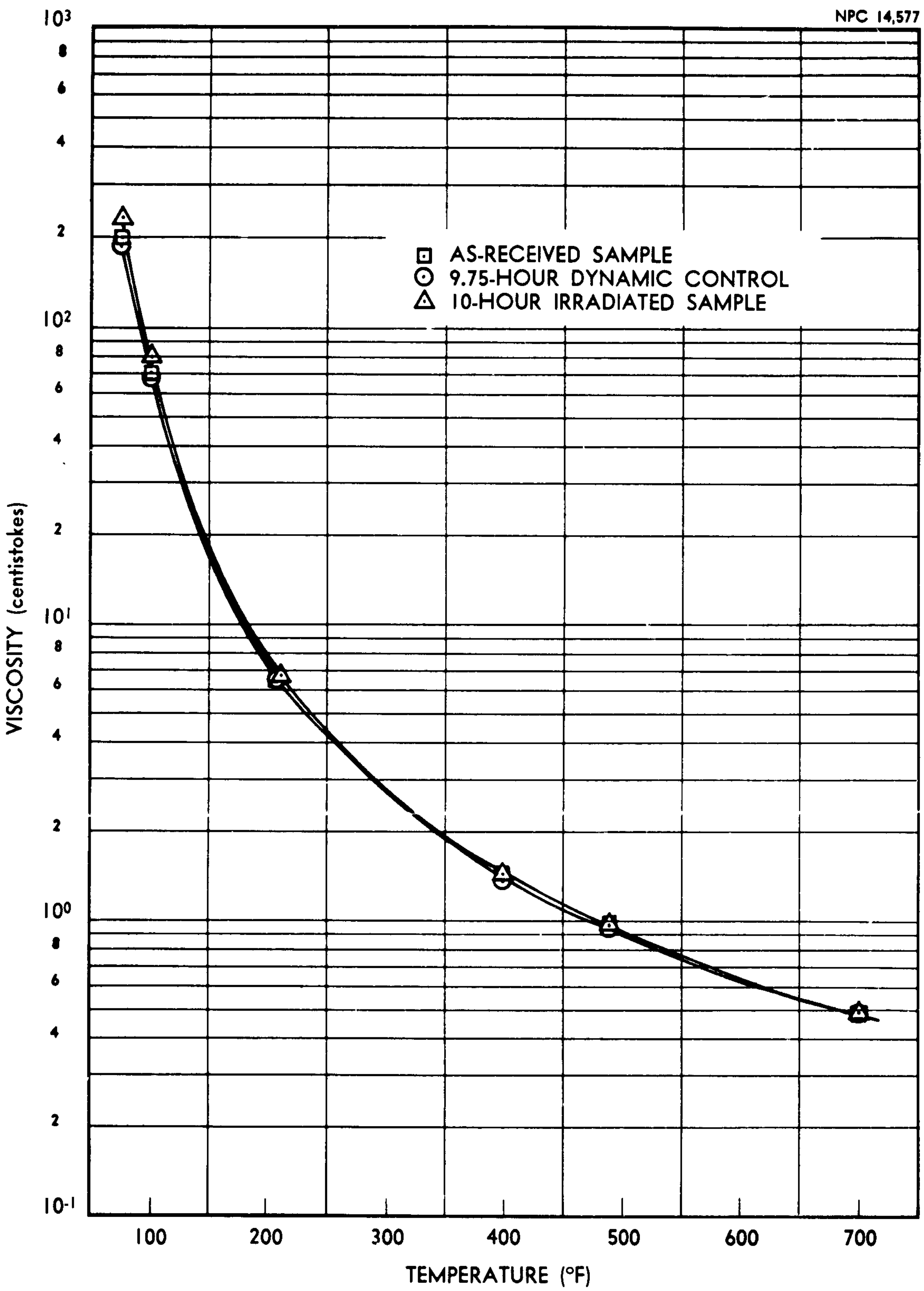

FIGURE 4. VISCOSITY VS TEMPERATURE FOR IRRADIATED AND NONIRRADIATED MIXED-4P3E

5.4 Fire Point and Autogenous Ingition Temperature

These data were very consistent and indicated no significant change due to irradiation.

5.5 Density

The very slight decrease evident in the irradiated sample was also evident in the dynamic control sample. No change due to irradiation was indicated.

5.6 Filtration

An increase in the particle weight above 5.0 microns was measured in the irradiated sample and in the dynamic control sample. Metal particles were evident in the dynamic control sample and are assumed to have contributed most of the weight increase in this sample. No metal particles were evident in the irradiated sample. Thus, it appears that the irradiation caused the formation of a significant quantity of insoluble particles above the 5.0-micron size.

5.7 Oxidation and Corrosion Stability

Complete oxidation-corrosion data are listed in Table III. Greater increases in neutralization number, viscosity, and copper corrosion in the irradiated sample indicate a slight loss of oxidation stability due to irradiation.

5.8 Lubricating Properties: Shell 4-Ball Wear

As determined in the 4-ball wear test (Table IV), the irradiation of Mixed-4P3E caused a slight loss of lubricating ability. Both the wear scars and coefficients of friction increased measurably. Since the viscosity data (Table II) indicated no change at the test temperature (400°F) and since the oxidation corrosion test at 400°F indicated no change in steel corrosion, the reason for this loss in lubricity is not evident. Significant changes in the boundary lubrication characteristics of a fluid can result, however, from very small changes in its chemical reactivity or film properties. It has also been shown (Ref. 5) that increased volatility adversely affects 4-ball wear. The vacuum distillation data indicate an increased volatility at relatively low temperatures for Mixed-4P3E.

5.9 ASTM Vacuum Distillation

The data from these tests are listed in Table V and plotted in Figure 5. The distillation data indicate that approximately 87% of the fluid was unchanged during irradiation, with about 9% being changed to less volatile forms and 4% to forms more volatile.

5.10 Vapor-Pressure-Temperature Characteristics

Vapor pressure was measured over the temperature range of from about 550°F to above the initial thermal decomposition point. Two determinations were made on the as-received fluid and two on samples

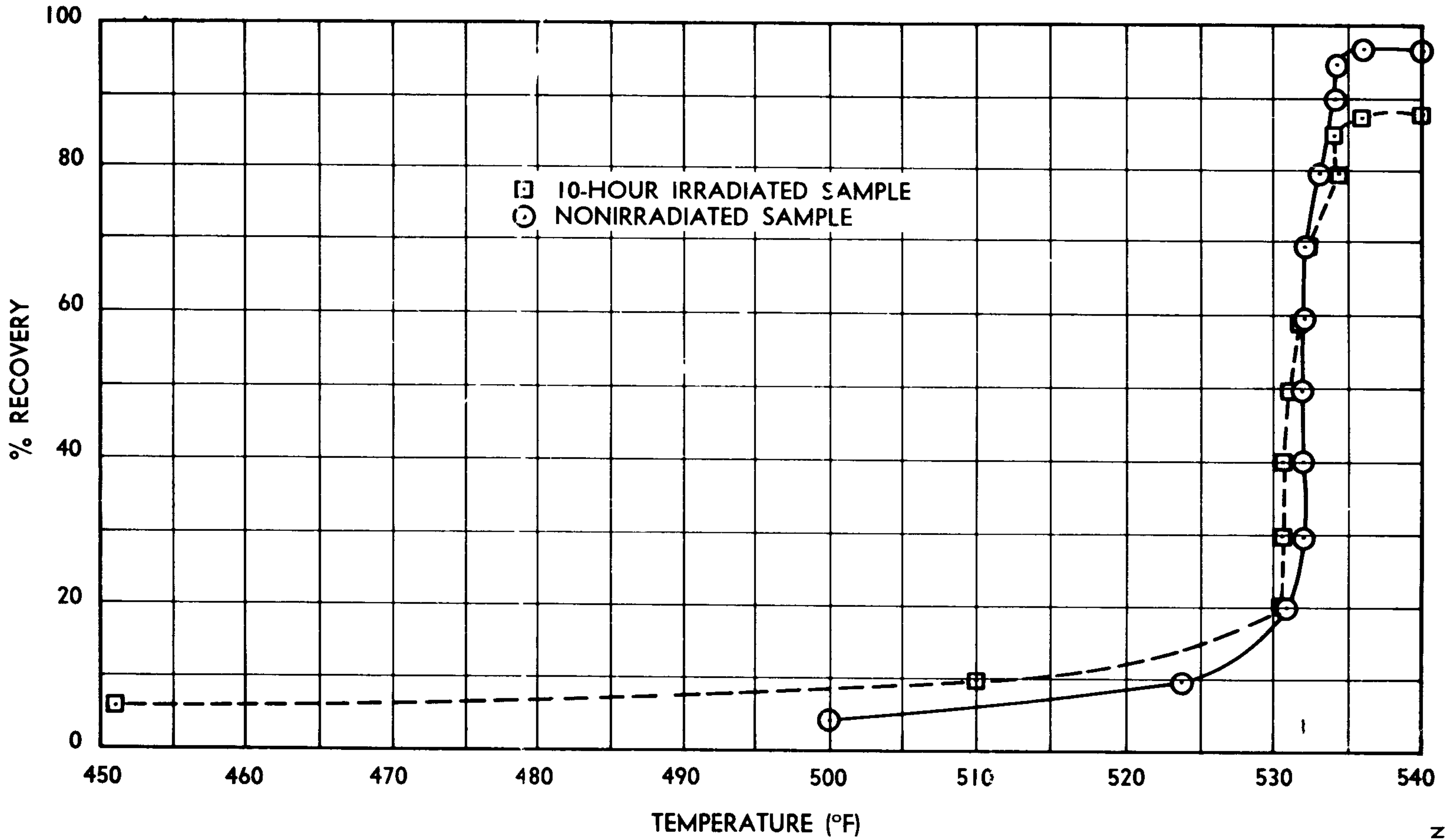

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FIGURE 5. ASTM VACUUM DISTILLATION OF IRRADIATED AND NONIRRADIATED MIXED-4P3E

from the 9.75-hour control run. Three determinations were made on the 10-hour irradiated fluid. Control data and irradiation data are listed in Tables VI and VII, respectively.

Shown in Figure 6 is the vapor-pressure curve for the second run on the as-received fluid. Points at 400, 450, and 500°F were measured in a separate experiment* but are included for completeness. Initial decomposition is indicated at approximately 837°F. This point was somewhat more distinct in the second run, but the data from both runs are in excellent agreement.

Figure 7 is the vapor-pressure curve for the second run on the 9.75-hour control sample. Decomposition is indicated at approximately 845°F. The data from both runs are in good agreement and indicate that the fluid was slightly more thermally stable after having been run 9.75 hours in the hydraulic loop.

Figure 8 is the vapor pressure curve for the third run on fluid from the 10-hour irradiated sample. An initial thermal decomposition point of about 815°F is indicated. This point is significantly different from the control data and indicates that some less stable products were formed during irradiation. The data from the second run are in good agreement with those plotted in Figure 8. During the first run, however, an additional pump-down was required, and it is felt that this deviation in procedure accounts for the inconsistent data.

*Performed in GD/FW's Test Laboratory and reported by H. J. Weltman in an internal document, FGT 2891 (26 October 1961).

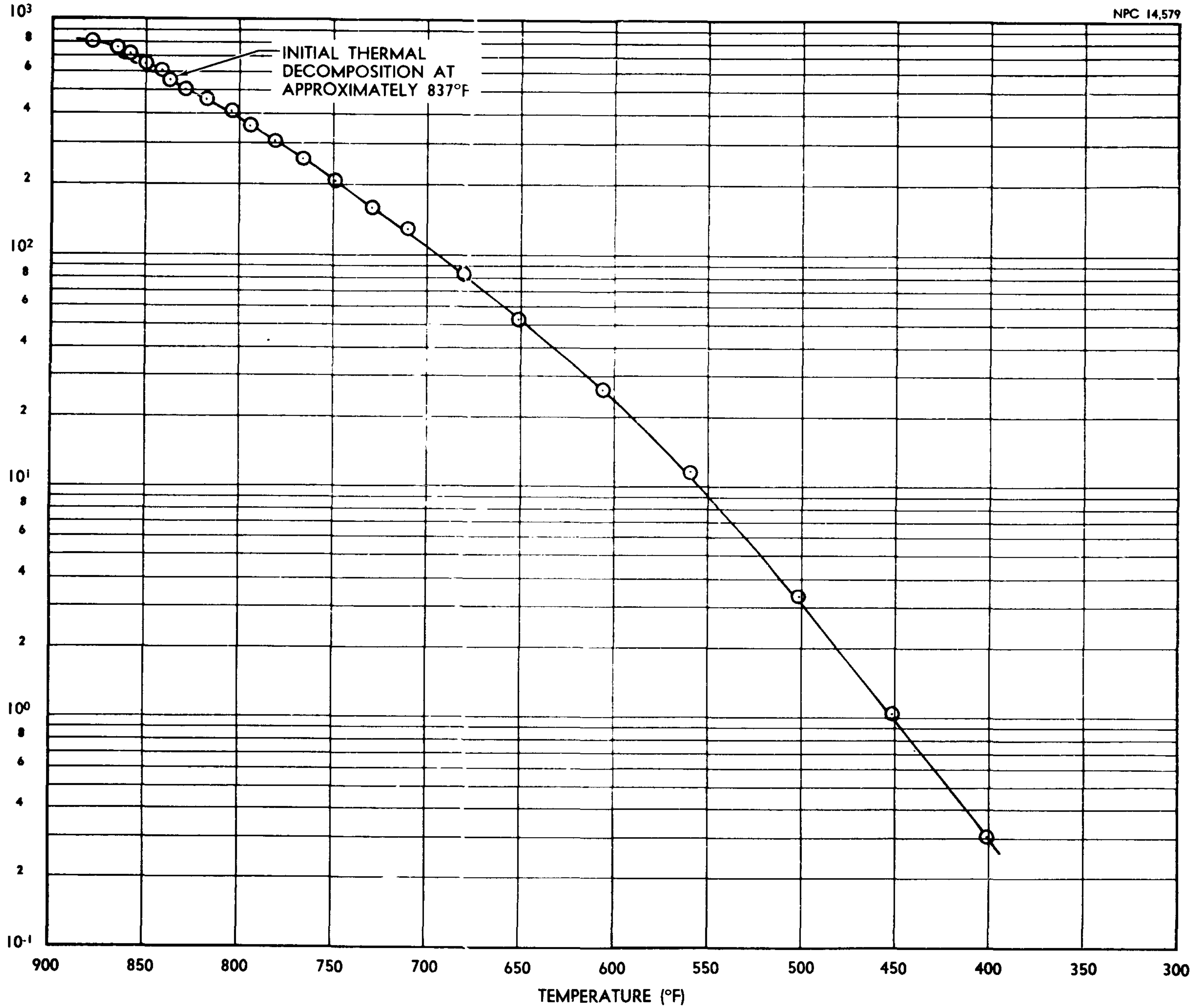


FIGURE 6. VAPOR PRESSURE VS TEMPERATURE FOR THE

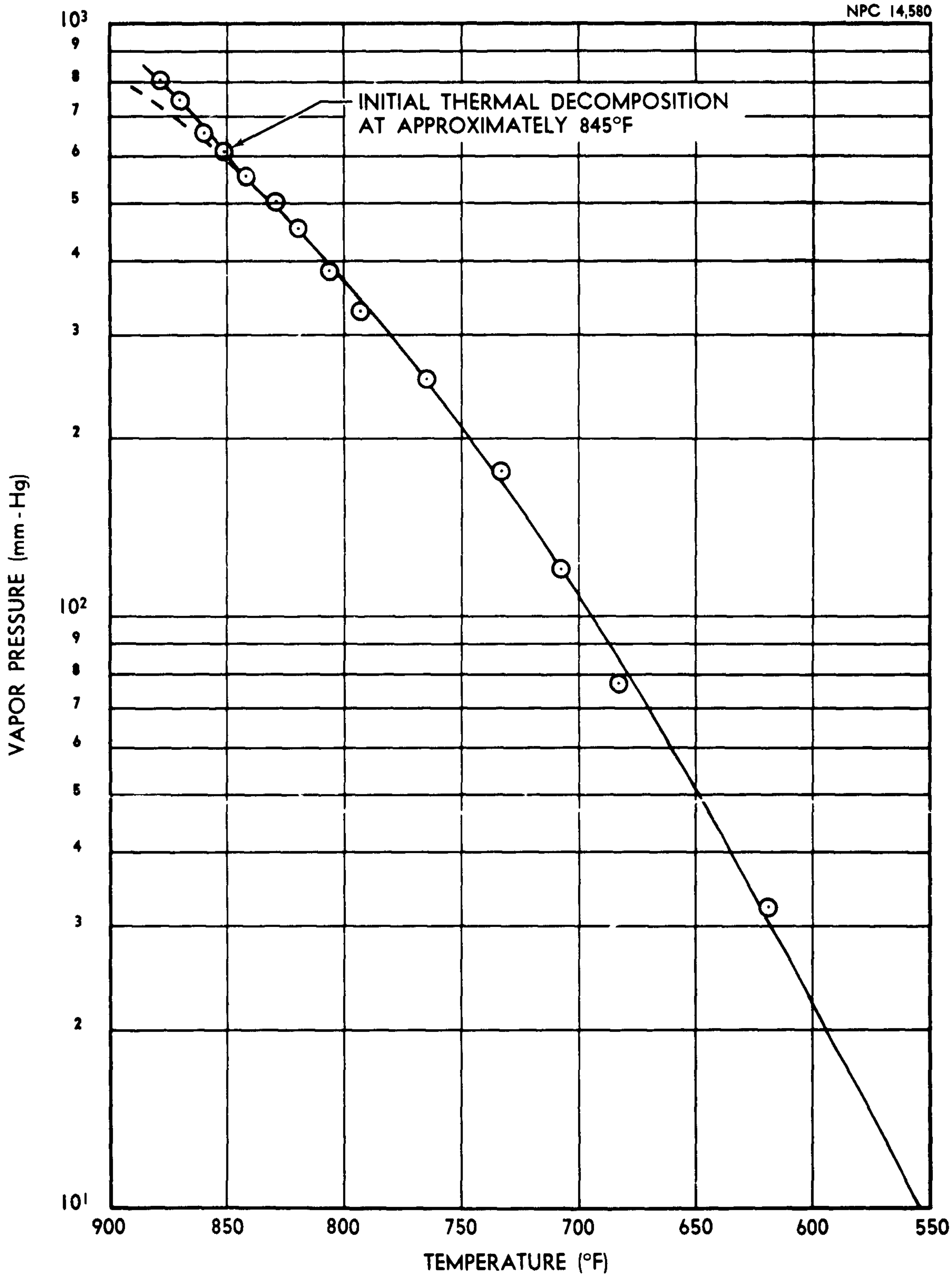


FIGURE 7. VAPOR PRESSURE VS TEMPERATURE FOR THE 9.75-HOUR DYNAMIC CONTROL SAMPLE OF MIXED-4P3E

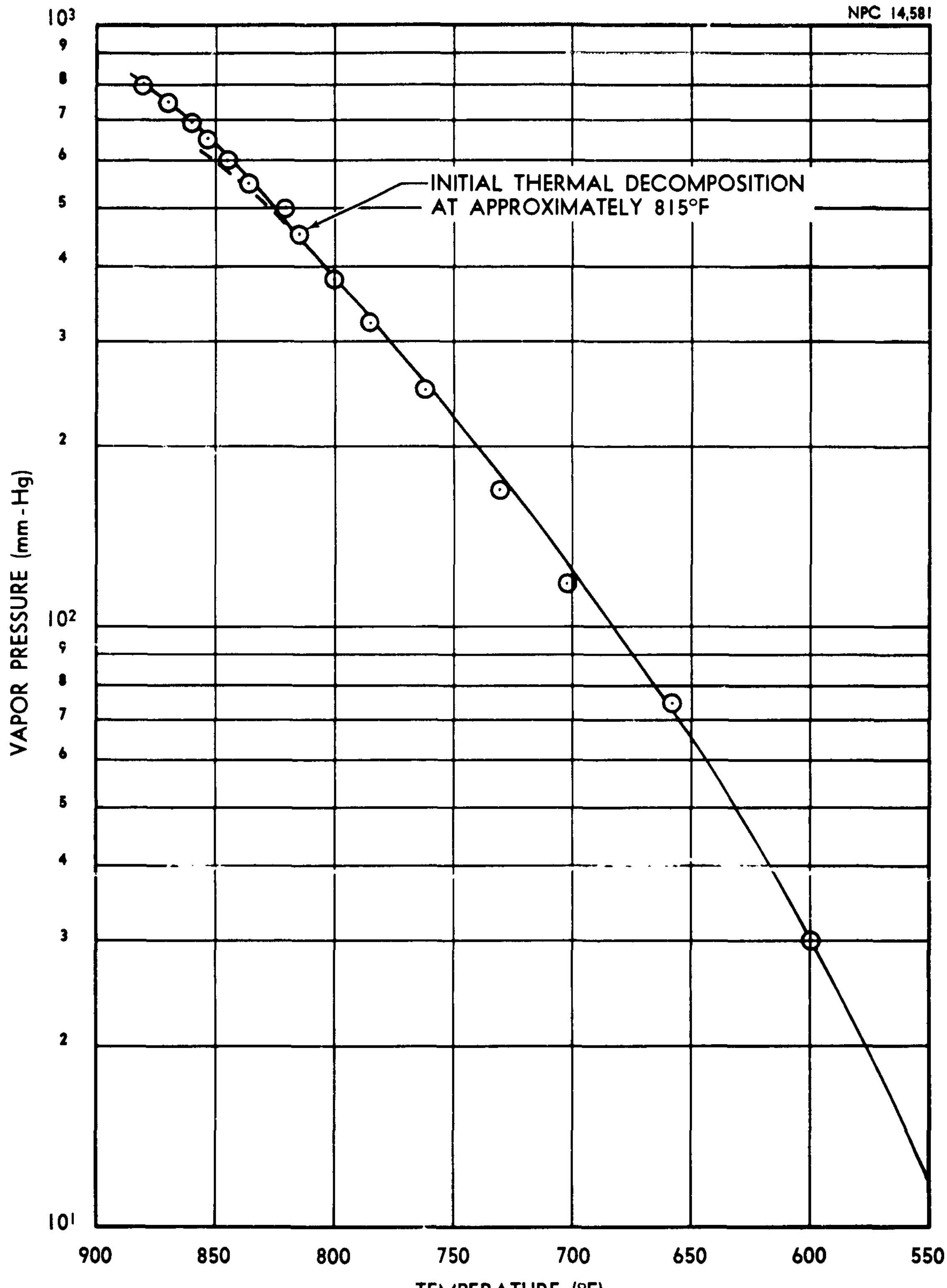


FIGURE 8. VAPOR PRESSURE VS TEMPERATURE FOR THE 10-HOUR IRRADIATED SAMPLE OF MIXED-4P3E (DETERMINATION NO. 3)

The vapor pressure curves shown in Figures 6, 7, and 8 are plotted as log pressure versus Fahrenheit temperature. The normal procedure for adjusting vapor pressure to a straight-line function is to plot log pressure versus the reciprocal of absolute temperature. This relationship is not exact, however, for large temperature ranges. Also, it was found that for these particular curves the initial thermal decomposition points were more difficult to determine from the straight-line plots.

VI. CONCLUSIONS

The following general conclusions represent the authors' overall interpretation of the data obtained in the experimental work covered by this report:

- The properties of Mixed-4P3E fluid were degraded only slightly during irradiation of the hydraulic pump loop. The conditions imposed - namely 3000 psi, 4 gpm, approximately $350^{\circ}F$, and 2.7×10^{10} ergs/gm(C) plus 3.9×10^{15} n/cm^2 - were not severe enough to be considered limiting conditions for this type of fluid.
- The important changes detected in Mixed-4P3E were increased viscosity, some loss of oxidation resistance, and the formation of constituents that were partly more volatile and partly less volatile than the original fluid. The fluid boiling range was broadened and some less thermally stable products were formed. Boundary lubrication properties were slightly degraded. Any of these changes could probably reach a limiting magnitude at higher radiation doses, higher temperatures, or more severe combinations of these environments.

APPENDIX

A-1 High-TemperatureViscosity Determinations

The apparatus used for viscosity measurements at elevated temperatures (490° and 704°F) consisted of a constant temperature vapor bath and a Cannon-Ubbelohde viscometer (size 25), as shown in Figure A-1. The Cannon-Ubbelohde-type viscometer was selected, because the viscometer constant is the same over the entire range of temperatures used in the test. Biphenyl was employed as the functional chemical for the 490°F vapor bath, and meta-terphenyl was used in the 704°F determinations.

Procedures for the viscosity determinations were in accordance with ASTM D445-53T. Temperature was monitored in the oil reservoir and alongside the efflux bulb. No significant differences were noted in temperatures at these points.

A-2 Neutralization Number Determination

Since phenols and phenolic compounds constitute a large percentage of the decomposition products of Mixed 4P3E fluids, neutralization numbers were determined not only by the standard ASTM D664-58 method but also by a special method used by Shell Development Company for the titration of phenols (Ref. 6).

The special method employs tetrabutylammonium hydroxide as the titrant and pyridine as the solvent. This method is described in detail in Reference 7.

In the series of tests conducted at this facility the titration endpoint was arbitrarily chosen as pH 11 instead of using the inflection point as specified in the special method. This decision was prompted by the pH 11 endpoint requirements of most of the current hydraulic fluid and lubricating oil military specifications.

A-3 Vapor Pressure and Thermal Stability Test

The apparatus employed for vapor pressure and thermal stability measurements on the test fluids was similar to the isoteniscope assembly described in Reference 8. A schematic diagram of the apparatus is shown in Figure A-2. A photograph of the setup is shown in Figure A-3.

The tests were conducted in the following manner:

1. The test fluid was introduced into the isoteniscope tube which was then placed inside the aluminum block heater and attached to the manifold assembly.

2. The assembly was then evacuated and the heaters turned on. Pumping was continued until the fluid temperature reached 200°F, at which time degassing was considered complete and stopcock 1 was closed. Heating was continued at a constant rate.

3. When refluxing became evident within the reservoir and reflux tube (observed through small window in aluminum block heater), equilibrium was considered reached and the fluid temperature and system pressure were recorded. Final pressure measurements were made with a Manostat manometer.

4. Nitrogen was then admitted to the system through stopcocks 2 and 3 until the desired pressure for the next measurement was reached. Step 3 was repeated.

5. Through repetitions of steps 3 and 4, measurements of vapor pressure vs. temperature were made over the desired ranges.

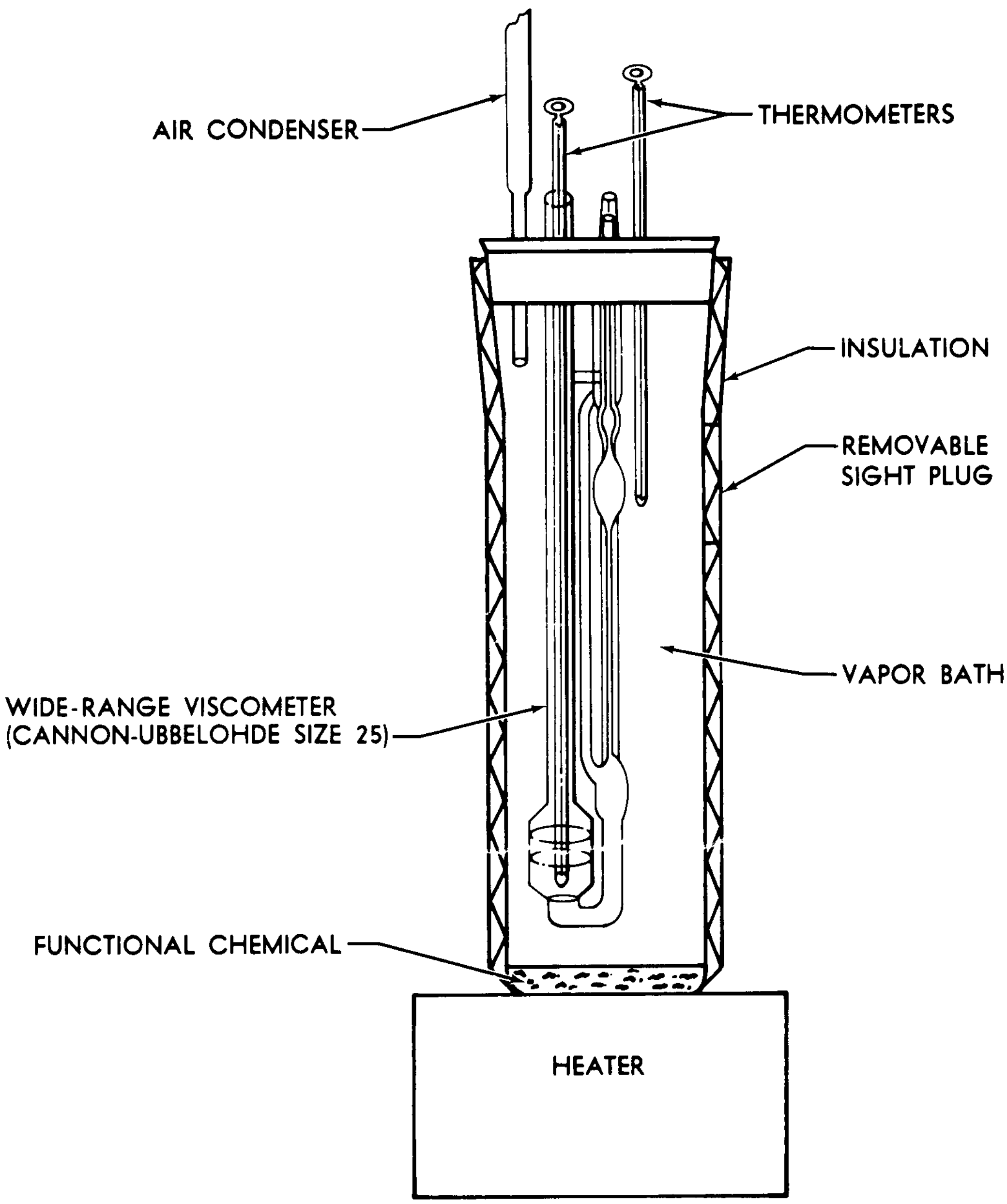


FIGURE A-1. SCHEMATIC DIAGRAM OF VERY-HIGH-TEMPERATURE VISCOSITY APPARATUS

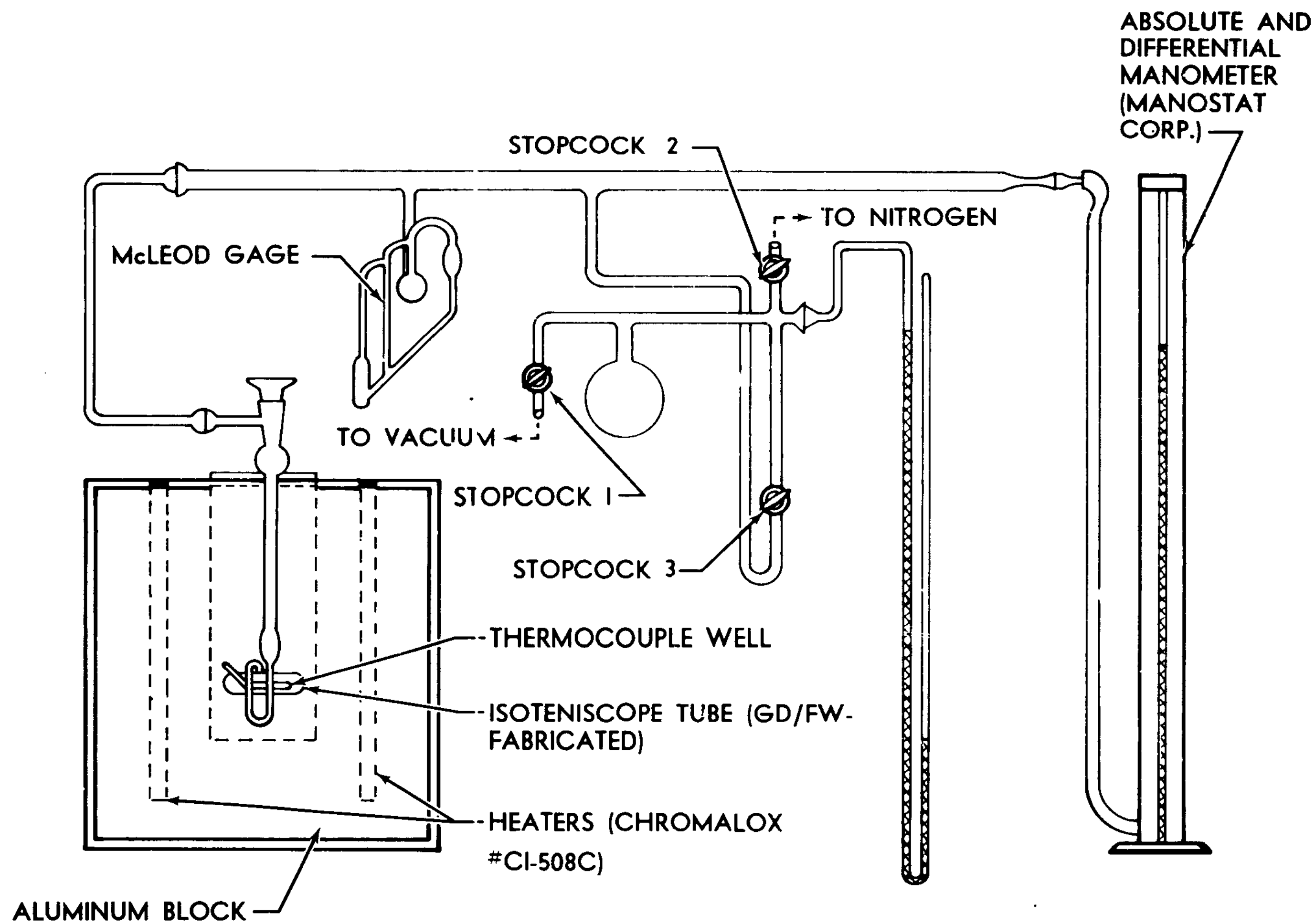
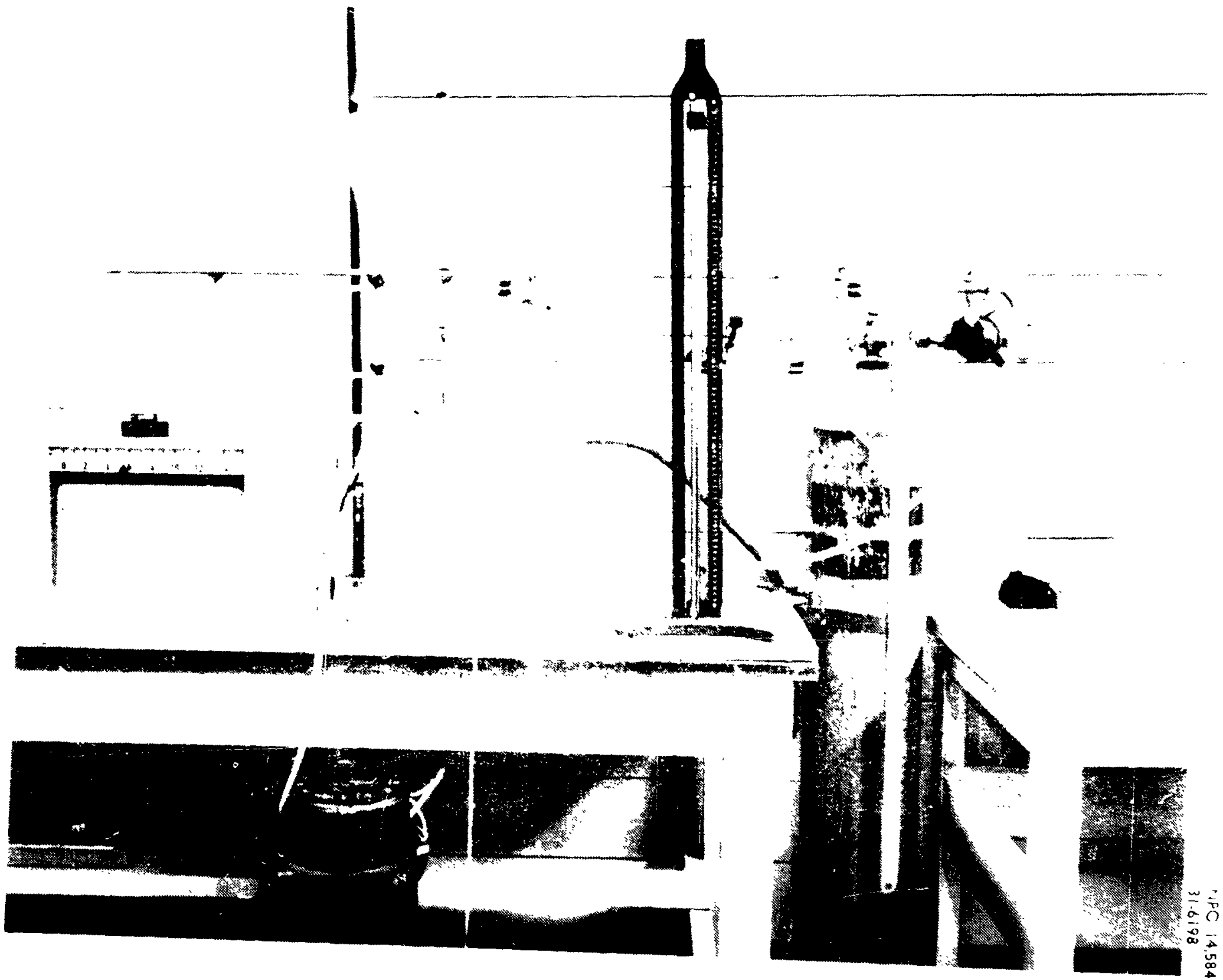


FIGURE A-2. SCHEMATIC DIAGRAM OF ISOTENISCOPE APPARATUS FOR VAPOR PRESSURE MEASUREMENTS



NPC 14,584
31-6198

FIGURE A-3. ISOTENISCOPE SETUP

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* All GD/FW reports published prior to July 1961 are referenced as Convair/Fort Worth reports

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(Contract AF 33(657)-7201) Unclassified report

An experimental hydraulic fluid, mix bis (phenoxyphenyl) ether, was irradiated under operational environmental conditions imposed by a hydraulic pump loop. The Ground Test Reactor at General Dynamics/Fort Worth was used to provide a mixed neutron-gamma radiation field.

The fluid was exposed to an average dose of 2.7×10^{10} ergs/gm(C) of gamma radiation and 3.9×10^{15} nvt of neutrons ($E_n > 2.9$ Mev). Pump-loop conditions during irradiation were 3000 psi and 4-gpm flow. Bulk-oil temperature ranged

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5. Ground Test Reactor (GTR)

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II. McDaniel, R. H.
III. Haley, F. A.
IV. Aeronautical Systems Division, Air Force Systems Command
V. Contract AF 33(657)-7201

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from 326 to 340°F.

A preirradiation control evaluation was conducted with the pump loop operating 9.75 hours and bulk-oil temperature ranging from 350 to 380°F.

The results of property and performance tests conducted on samples of the fluid during irradiation were compared to data on the nonirradiated fluid to assess the radiation damage. All fluid testing was accomplished in the GD/FW Irradiated Materials Laboratory.

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